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13. ABSTRACT (Maximum 200 words)  Four Institutes in Russia were supported for the current year. Technical areas include high-temperature electronics with diamond-like carbon materials, laser resonance photoelectron and photoion microscopy, near-field laser microscopy, alteration of the optical properties of materials, atom lithography with laser light, tunable laser diode spectroscopy, remote sensing from space, and UV and X-Ray optics.				
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**FINAL REPORT**

**May 1998-April 1999**

**High-Powered CO<sub>2</sub> Laser  
Interactions with Surfaces  
And  
Related Studies**

**Grant #DAAH04-95-1-0344**

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## **Executive Summary**

Four Institutes in Russia were supported for the current year. Technical areas include high-temperature electronics with diamond-like carbon materials, laser resonance photoelectron and photoion microscopy, near-field laser microscopy, alteration of the optical properties of materials, atom lithography with laser light, tunable laser diode spectroscopy, remote sensing from space, and UV and X-Ray optics.

Exceptional productivity was achieved resulting in 85 publications at a cost of less than \$8K per paper produced. This cost-based productivity measure is an order of magnitude higher than that achieved by any U.S. institution.

The quality of the work is considered at least comparable to most large U.S. research establishments, with selected citation index analysis revealing better averages than the journals of the Optical Society of America, for example, and much higher than SPIE publications or the journal Infrared Physics.

A technology transfer project separately funded by the White House Office of Drug Control Policy was begun this year. The project investigates the advantages of infrared and tunable laser spectroscopy for drug detection at border crossing, in airports, and in broad-area search.

## **Technology Transfer and Quality**

One of the many ways in which the success of attaining the technology transfer goal can be measured is in the influence of the published papers on the work of others. This influence can be measured using the Science Citation Index® which is published by the Institute for Scientific Information® (ISI®). We have used the ISI Citation Index to examine the work of just two of the Russian scientists who are supported by the program. Since 1992, V.S. Letokhov has published 97 papers which have been cited in 454 papers by other workers. Also since 1992, A. M. Prokhorov has published 60 papers which have been cited 347 times. These data imply an "impact factor" (number of citations per paper) of 5.78 for Dr. Prokhorov and 4.68 for Dr. Letokhov. To appreciate the quality of

these numbers, here are average impact factors for articles published in a few example journals:

<i>JOSA A</i> .....	1.425
<i>JOSA B</i> .....	2.157
<i>Applied Optics</i> .....	1.033
<i>Phys. Rev. A</i> .....	2.292
<i>Phys. Rev. B</i> .....	3.187
<i>Phys. Rev. C</i> .....	1.842
<i>Phys. Rev. D</i> .....	3.233
<i>Phys. Rev. E</i> .....	1.888
<i>Optical Engineering</i> .....	0.65
<i>Infrared Physics and Technology</i> ...	0.483

A technology transfer project separately funded in excess of \$500K by the White House Office of Drug Control Policy was begun this year. The project investigates the advantages of infrared and tunable laser spectroscopy for drug detection at border crossings, in airports, and in broad-area search.

## **Institutes and Technical Areas**

Each of the Institutes is listed with a description of the work supported under the program. A highly detailed report on the work supported at the A.F. Ioffe Physico-Technical Institute St. Petersburg, Russia is contained in Appendix A.

### **I. Institute of Spectroscopy of Russian Academy of Sciences Laser Spectroscopy Department Troitsk, Moscow Region, 142092 Russian Federation**

Our main research supported by DOD/UOA are in the field of NANOOPTICS.

Nanooptics is a new trend of research and development in nanoscience and nanotechnology, which is based on the observation

and application of new effects of the interaction (linear and nonlinear) between laser light and materials (atoms, molecules, surfaces, etc.) on a spatial scale much smaller than the laser wavelength. It is usually believed that such a formulation of the problem is nonsensical because the laser light cannot be localized (e.g., focused) in a space smaller than its wavelength. In actual fact, when using sub-micron structures (nanoscales, nanotips, nanostrips etc.), light can be confined within smaller volumes of space in the same way as in the case of microwave radiation. However, as distinct from the microwave case, of great importance in the optical case become the atomic-molecular (discrete) structure and quantum properties of the substance interacting with such a localized light. Hence follow two fundamental properties:

- first, the use of highly localized laser light enables one to study the structure of the substance with a nanometer-high spatial resolution;
- secondly, the response of the substance to localized light changes materially in comparison with the case where it interacts with a propagating light field.

Investigations into these new capabilities of nanooptics is the prime objective of the Lab. of Laser Spectroscopy of the Institute of Spectroscopy of the Russian Academy of Sciences, Moscow, Russia.

They have at least two potentially important practical applications:

- (1) the laser spectromicroscopy of materials with a nanometer-high spatial resolution and chemical selectivity and
- (2) an optical memory with a ultrahigh density (of the order of 1000 Gbt per square inch).

The main themes of investigations are as follows:

- (1) **LASER RESONANCE PHOTOELECTRON and PHOTOION MICROSCOPY** with a spatial resolution of 3-30 nm. The development of a microscope using excitation with laser pulses, femtosecond pulses included, which make it possible to and select optical fiber nanotips with

a single excitable light absorbing center for further use as a nanoprobe of molecular structures, surfaces, etc. The same microscope is utilized in experimental studies into the possibility of developing a ultrahigh-density WROM;

- (2) **NEAR-FIELD LASER MICROSCOPY** with a lateral spatial resolution of some 30 nm and a longitudinal resolution better than 1 nm. In contrast to the existing near-field microscopy schemes, the microscope developed provides for the exact measurement of the distance between the nanotip surface and the sample under study. This makes it possible to investigate the processes of resonance interaction between molecules (resonance energy transfer) as a function of their spacing. This in turn forms the basis for the implementation of the chemically selective probing of surfaces by means of a fiber optical nanotips with a single photoexcitable center for taking measurements with a nanometer-high spatial resolution. This type of microscope will open up entirely new instrumental possibilities for nanoscience and nanotechnology;
- (3) **ALTERATION of the OPTICAL CHARACTERISTICS of MATERIALS** (atoms, molecules) in a nanoscale, i.e., vicinity to and inside nanostructures, nanotips and nanospheres in particular. The subjects of current studies include changes of resonance frequencies and probabilities of radiative transitions in atoms, especially forbidden atomic transitions which make it possible to induce nanolocal modifications in the properties of materials for numerous applications (microscopy, optical memory, etc.).

This research is a natural extension of our research in 1996-1997 supported by DOD/UOA in the field of:

- (4) **ATOM LITHOGRAPHY WITH LASER LIGHT.** Rapid progress of methods of laser control of atom motion allows us to realize an entirely new approach of control of atomic and (potentially) molecule beam. Particularly, it becomes possible to collimate, reflect and focus the atom beam. Laser-induced focusing of atomic beam can be very efficient to allow us to achieve the size of a focusing spot about a few angstrom, i.e. less than 1nm. Ultimate spatial resolution of atom optics is determined by size of an atom and its Broglie wavelength. It will be important to realize these possibilities in experiment.

We considered the following two approaches in a deep focusing of atomic (molecule) beams.

The first one is based on using evanescent wave as an atom concave mirror. When a plane traveling light wave is totally reflected internally at the surface of dielectric in a vacuum, a thin evanescent wave is generated on the surface. It is this surface wave that can be served as an atomic mirror for an atom running into it: with a positive laser frequency detuning, the gradient force expels atoms out of the field. The shape of dielectric surface determines a focusing properties of atom concave mirror.

Another approach will explore the near-zone field laser light configuration. Consider an intense plane electromagnetic wave incident normally on a small circular aperture of radius  $a < \lambda$  in conducting plate. The near-zone field of this small aperture can act on the moving atom through is as an atom lens. Both considered configurations permit to reach atomic diffraction resolution in the focusing of atomic (molecule) beams (1-10 nm).

All these avenues of inquiry are combined under the name of **NANOOPTICS** as part of R/D in the **field of nanoscience and nanotechnology**.

## **II. Applied Tunable Diode Laser Spectroscopy at the General Physics Institute, Moscow, Russia.**

The new high sensitive and high selective device for detection of trace impurities concentrations in open atmosphere was developed. The device features:

- near infrared laser and detector, operating at room temperatures,
- the unique "Chernin" multipass cell with up to 500 passes over 50cm - the base distance between two sets of mirrors,
- quick pulsed method of measurements, which allowed to increase signal-to-noise ratio by suppressing low frequency laser noise,
- the original data acquisition system, based on comparison of the recorded analytic portion of spectrum with reference spectrum, obtained simultaneously,

complete automated operation of the chosen impurity monitoring in open atmosphere with storing of the recorded data in computer memory.

The device was applied for detection of methane in the open atmosphere, diode laser with wavelength 1.65  $\mu\text{m}$  was used. Sensitivity of 50 ppb was achieved, which is enough for monitoring of methane in the atmosphere (1.7 ppm in clean air).

The same device was applied for the detection of ethanol vapor in the atmosphere and in the air samples. The ethanol was chosen as a model molecule with which the technique of tunable diode laser spectroscopy application for the detection of complex organic molecules, such as drugs, should be tested. It was taken into account that precise and selective detection of ethanol in air samples might have its own significance and might be applied for the alcohol tests in driver's breath.

The alcohol molecule is more complicated than methane and its spectrum generally consist of broad bands instead of narrow separated lines as in the case of methane and others simple molecules. Preliminary registration of ethanol vapor absorption spectrum was performed using the FTIR spectrometer Bruker 113-v and spectral region for diode laser was chosen near  $7070\text{ cm}^{-1}$ , which contains finger-like Q-branch (first overtone of O-H stretch). DFB diode laser for this region (Sensors Unlimited, US) used in mode hopping regime, allowed to get sensitivity 50 ppm over 3 m optical path, which is equivalent to 20 mg/210 l (the unit, accepted as permitted level of ethanol vapor concentration in human breath).

The obtained results suggested not only the possibility of manufacturing of the high selective device for detection of alcohol in the human breath, but the possibility of developing the device for distant detection of alcohol vapor inside the moving car. The laboratory prototype of the device was demonstrated at the Optical Sciences Center of the University of Arizona in November - December 1999.

The infrared spectrum of  $\text{C}_9\text{H}_{10}\text{O}$  (Phenol, 2-(2-propenyl-), P2P) was investigated. This substance is one of sub-products appearing during underground preparation of drugs, and could be used as indicator of such illegal production. Its absorption spectrums in liquid and gaseous states in the wavelength range 2-12  $\mu\text{m}$  have been measured. The absorption spectra of a gas phase were recorded at various temperatures in the range 25-75 C. Fitting with library gas-phase spectrum (National Institute of



Standards, USA) was carried out. The analysis of temperature dependencies of absorption peaks allowed to detect impurity with higher saturated vapor pressure compared to the main substance. The peaks of this impurity were found in the library spectrum too.

The experiments on ethanol vapor measurement by a method of Fourier-spectrometry were carried out. The experimental setup consisted of Bruker infrared Fourier-spectrometer IFS-66 v/s, multipass optical cell with a variable optical path length (0.8-8 m) and dosing system permitting to create necessary concentration of ethanol in the cell. The achieved detection limit was about 1 ppm. Such ethanol concentration in human exhalation corresponds to ethanol concentration in blood of approximately  $5 \cdot 10^{-3}$  gram per liter. This level is two orders of magnitude lower than the permitted level for driving a vehicle.

#### Fundamental researches in diode laser physics

The investigations of spectral lines pressure broadening and shift in the mid-IR region were continued. Significant attention was paid to provide high accuracy of line shape measurements and to study sources of errors limiting the accuracy of diode laser absorption spectroscopy. Instrumental function of diode laser spectrometer was studied in details. One of the parameters which has a significant influence to the accuracy of line shape recording is so called "optical zero" level, i.e. a level of zero transmission. Our studies of a diode laser instrumental function allow to solve a problem of optical zero, and to perform a correct line shape fitting with accuracy better than 1 %. The instrumental function of a distributed feedback diode laser operating in mid-IR region can be determined as a product of the two Lorentzian functions with spectral widths of about 60 MHz and 10 GHz.

#### Thermophysics Spectroscopy

The method of Temperature-Waves-Transmission Spectroscopy is developed on the basis of the classical AC calorimetry technique. The idea of the method is to use the information about the phase and the amplitude of the temperature wave transmitted through a plate-like sample for simultaneous determination of the sample's heat capacity and thermal conductivity. For the correct application of the method in an experimental

set up, it was necessary to study the problem of a plane temperature wave transmission through a plate-like multilayered system. This problem was solved analytically for a wide range of experimental conditions. It was shown that the AC calorimeter can be used at relatively high frequencies, when the temperature oscillations in the sample are not quasi-static. Therefore, the width of appropriate frequency range of the classical AC calorimetry can be enlarged. This is of great importance for applicability of classical AC calorimetry for low heat-conductive materials, such as polymers. It is shown that dynamic heat capacity and thermal conductivity of polymers can be measured in real time and in broad frequency region. The method was applied for polystyrene near glass transition and for 4,4'-n-octyloxybiphenyl (8OCB) liquid crystal in melting region, smectic-nematic and nematic-isotropic transitions. The possibility of dynamic heat capacity measurements is demonstrated.

The advanced AC technique was applied for investigation of the melting kinetics in polycaprolactone. It was found that melting in polycaprolactone is related to an activated process. The activation of the melting process after a step heating is described by a stretched exponent and the decay of the melting by only one exponent at short times. The dependencies of the exponent on temperature and thermal treatment were investigated at frequencies in the range 0.1-1Hz and temperature-modulation amplitudes 0.005-0.2K.

### **III. A.F. Ioffe Physico-Technical Institute St. Petersburg, Russia.**

A highly detailed report on the work supported here is contained in Appendix A. The area of high temperature materials for electronics is a critical one for applications which require high power dissipation by solid-state devices. Diamond-like carbon materials are being developed for efficient heat transfer and high temperature tolerance in high power amplifiers, for example.

### **IV. The Lebedev Physical-Technical Institute, Moscow.**

Instrumentation for remote sensing, including space qualified instruments, are developed at the Lebedev Institute. Applications of the instrument technology within the laboratory is also pursued, resulting in applications for monitoring the formation of thin films.

## **Productivity**

Here we list the publications of each institute supported during any part of the year. Only work supported by the project published during the performance period is reported. The productivity rate of less than \$8K per paper is an order of magnitude higher than can be achieved in the U.S.

### **I. Reports on Period 1.01.1998-31.12.1999 of Laser Spectroscopy Department, Institute of Spectroscopy, Troitsk**

During 1998 year 16 papers in refereed journals: 11 in International Journals (Phys. Rev., J. Mod. Opt., Opt. Comm. etc.) and 5 Russian Journals (JETP Lett. and Proceed. of Russian Academy of Sciences were published. List of papers is following:

- 1) Near Field Diffraction Grating for Atoms. V.I.Balykin, D.A.Lapshin, M.V.Subbotin, V.S.Letokhov. Optics Comm., 145, 322-328 (1998).
- 2) Statistical Inhomogeneous Broadening of Infrared and Raman Transitions in Highly Vibrationally Excited XF6 Molecules. A.A.Makarov, I.Yu.Petrova, E.A.Ryabov, V.S.Letokhov. Journ. Phys. Chem. A102, 1438-1449 (1998).
- 3) Contact Scanning Optical Near-Field Microscopy. D.A.Lapshin, V.N.Reshetov, S.K.Sekatskii, V.S.Letokhov. Pis'ma Zh. Eksp. Teor. Fiz. 67, N4, 245-250 (1998) (in Russian).
- 4) On the Possibility of Ultra-High Density WROM Based on the Using of Laser and Corpuscular Beams. V.S.Letokhov, S.K.Sekatskii. Optics Comm. 147, N1-3, 19-25 (1998).
- 5) Emission Spectroscopy of a Carbon Plasma Formed by Laser Ablation of Graphite. 2. Ablation by a CO2 Laser and also Simultaneously by XeCl and CO2 Lasers. A.V.Dem'yanenko, V.S.Letokhov, A.A.Puretskii, E.A.Ryabov. Kvantovaya Elektronika 24, N1, 36-40 (1998).
- 6) Imaging of the Near-Field over the Reflection Phase Grating by the Apertureless Photon Scanning Tunneling Microscope. D.A.Lapshin, V.I.Balykin, V.S.Letokhov. Journ. of Modern Optics 45, 747-758 (1998).

- 7) Resonance Energy Exchange at Nanoscale Curved Interface.  
V.V.Klimov, V.S.Letokhov. Chem. Phys. Lett. 285, 313-320 (1998).
- 8) IR Multiple-Photon Dissociation of Br (CF<sub>2</sub>)<sub>4</sub>COI Molecules by  
Excitation via C=O (1794 cm<sup>-1</sup>) Stretching and Sceleton (961 cm<sup>-1</sup>)  
Vibrations. A.V.Dem'yanenko, E.A.Ryabov, V.S.Letokhov. Chem. Phys.  
Lett. 286, 277-283 (1998).
- 9) Fiberoptic Evanescent mid-IR Fourier Transform Spectroscopy of  
Cancer Tissue. N.Afanasieva, V.Artjushenko, S.Kolyakov, A.Lerman,  
D.Pakhomov, V.Sokolov, V.Letokhov. Lasers in the Life Sciences 8, N2,  
65-79 (1998).
- 10) Photoselective Laser Photoion Microscopy with 5nm Resolution.  
S.K.Sekatskii, D.V.Serebryakov, V.S.Letokhov. Pis'ma Zh. Eksp. Teor. Fiz.  
67, N7, 450-454 (1998), [JETP Lett. 67, N7, 470-475 (1998)].
- 11) Femtosecond Two-Photon Laser Photoelectron Microscopy.  
S.K.Sekatskii, S.V.Chekalin, A.L.Ivanov, Yu.A.Matveets, A.G.Stepanov,  
V.S.Letokhov. Journ. Phys. Chem. A102, 4148-4153 (1998).
- 12) Resonance Fluorescence of System Atom + Dielectric Microsphere,  
excited by Single Photon. V.V.Klimov, V.S.Letokhov. Pis'ma ZhETF 68,  
115-120 (1998); [JETP Lett. 68, 124-130 (1998)].
- 13) Resonance Interaction between Two Atomic Dipoles Separated by the  
Surface of a Dielectric Nanosphere. V.V.Klimov, V.S.Letokhov Phys. Rev.  
A58, 3235-3247 (1998).
- 14) Transition Spectra in the Vibrational Quasicontinuum of Polyatomic  
Molecules. Raman Spectra of Highly Excited SF<sub>6</sub> Molecules.  
A.L.Malinovsky, I.Yu.Petrova, E.A.Ryabov, A.A.Makarov, V.S.Letokhov.  
Journ. Phys. Chem. 102, 9353-9359 (1998).
- 15) Direct Observation of Light-Absorbing Nanocrystals in Glass Matrixes  
by the Projection Laser Photoion Microscopy Technique. S.K.Sekatskii,  
D.V.Serebryakov, V.S.Letokhov. Rev. Sci. Instr. 69, N11, 3885-3888  
(1998).
- 16) Effect of the Charge and Position of Atom on Parity Violation Energy  
Difference of Molecular Enantiomers. L.N.Ivanov, V.S.Letokhov.  
Proceedings of Russian Academy of Sciences 362, N1, 36-39 (1998).

We presented during 1998 our results of following International Conferences:

- (1) International Symposium on Modern Physics, 15-16 May, 1998, Heidelberg, Germany. Invited talk by V.Letokhov with title: "Laser Photoelectron/Photoion Microscopy with Subwavelength Spatial Resolution (3-30nm)";
- (2) XXIV European Congress on Molecular Spectroscopy, Prague, Czech Republic, August 23-28, 1998. Invited talk by V.Letokhov with title "Molecular Enantiomers and Laser Spectroscopy";
- (3) EUROPE EQEC/CLEO, Glasgow, UK, 14 -18 September, 1998. Invited talk by V.Letokhov with title "Optical Tomography";
- (4) NATO Research Conference "Quantum Optics 98", 29 September – 4 October, 1998, Piza, Italy. Invited talk by V.Letokhov with title: "Atom Confinement and Optics: from micro- to nanoscale".

## **II. Applied Tunable Diode Laser Spectroscopy at the General Physics Institute, Moscow.**

- 1) A.Berezin, S.Chernin, O.Ershov, V.Kutnyak, A.Nadezhdinskii "Portable methane analyzer based on tuned near IR diode laser for measurements in open atmosphere.", Proceedings of 2nd International Conference on Tunable Diode Laser Spectroscopy, Moscow, 1998, p.33.
- 2) A.Berezin, Yu.Bugoslavskii, O.Ershov, V.Kutnyak, A.Nadezhdinskii "Measurement of Ethanol concentration with near IR Diode Lasers", Proceedings of 2nd International Conference on Tunable Diode Laser Spectroscopy, Moscow, 1998, p.34.
- 3) S.M.Chernin "New promising multipass systems for FTIR and TDL spectroscopy", Proceedings of 2nd International Conference on Tunable Diode Laser Spectroscopy, Moscow, 1998, p.35.
- 4) P.Gibson, D.Rivin, A.Berezin, O.Ershov, V.Kutnyak, A.Nadezhdinskii "Application of diode lasers for measurement of water vapor diffusion through laminated fabrics and membranes", Proceedings of 2nd

International Conference on Tunable Diode Laser Spectroscopy, Moscow, 1998, p.36.

5) V.Plotnichenco, Yu.Pyrcov, M.Slipchenco, O.Ershov, A.Nadezhdinskii, M.Spiridonov "Current and temperature dependence of emission characteristic of semiconductor lasers measured by Fourier Transform Spectroscopy", Proceedings of 2nd International Conference on Tunable Diode Laser Spectroscopy, Moscow, 1998, p.32.

6) A.Nadezhdinskii, M.Spiridonov "Air broadening and shift of acetylene in 1.5 mkm region.", Proceedings of 2nd International Conference on Tunable Diode Laser Spectroscopy, Moscow, 1998, p.31.

7) A.D.Britov, A.I.Nadezhdinskii, A.G.Berezin, A.S.Kononov, N.A.Suleymanov, O.V.Ershov, V.G.Kutnyak, "Medical gas analyzer on near IR lasers and detectors" Proceedings of XV International conference on optoelectronics, electronic and ion-plazma technology, Moscow, 1998, p. 89, in Russian.

8) A.Nadezhdinskii, A.Berezin, S.Chernin, O.Ershov, V.Kutnyak "High sensitive methane analyzer based on tuned near IR diode laser." Spectrochimica acta part A, 1998, to be published.

9) A.Nadezhdinskii, A.Berezin, Yu.Bugoslavskii, O.Ershov, V.Kutnyak "Application of near IR Diode Lasers for Measurement of Ethanol vapor", Spectrochimica acta part A, 1998, to be published.

10) S.M.Chernin "Development of multipass matrix systems", Spectrochimica acta part A, 1998, to be published.

11) P.Gibson, D.Rivin, A.Berezin, A.Nadezhdinskii "Measurement of water vapor diffusion through laminated fabrics and membranes using a diode laser spectroscope.", U.S. Army Natick Research, Development, and Engineering Center Technical Report, Natick/TR-98/004, March, 1998.

12) M. V. Spiridonov. "Diagnostic of CO2 laser active media using tunable diode laser spectroscopy technique". International conference on CO2 lasers Physics and Applications, p.17, Gdansk, Poland, 1998.

13) A.A.Minakov, Yu.V.Bugoslavsky, C.Schick "Improvement of AC calorimetry for simultaneous measurements of heat capacity and thermal conductivity of polymers", Thermochemica Acta, v 317, 1998, pp.117-131.

14) A.A.Minakov, C.Schick "Advanced AC calorimetry of polycaprolactone in melting region", to be published in *Thermochimica Acta*.

15) A.A.Minakov "AC calorimetry of polymers in glass and melting transitions", V Lahnwitzseminar on calorimetry, 7-12 June, 1998, Kuhlungsborn, Germany.

16) Bugoslavsky Y.V., Emelchenko G.A., Caplin A.D. "Influence of surface barriers on the magnetization of ultra-fine powders of Y-Ba-Cu-O", 5th international workshop on physics and applications of high-temperature superconductors, MSU, Moscow, April 1998.

17) C.Schick, U.Jonsson, T.Vassiliev, A.Minakov, J.Schawe, R.Scherrenberg and D.Lorenczy, Applicability of 8OCB for Temperature Calibration of Temperature Modulated Calorimeters, *Thermochimica Acta*, in print.

18) A.A.Minakov and I.Hatta, Thermal contact conductance in advanced AC calorimetry, *Thermochimica Acta*, in print.

19) A.A.Minakov, Advanced AC calorimetry for simultaneous measurements of dynamic heat capacity and thermal conductivity of polymers, NATO Advanced Research Workshop on "Polymer Structure and Transport in Confined Spaces" Web.page is available:  
<http://www.carb.nist.gov/biotech/NATO99/index.html>

### **III. Lebedev Institute, Moscow, Russia.**

1) I. Zhitnik, A. Ignatiev, V. Korneev, V. Krutov, S. Kuzin, A. Mitrofanov, S. Oparin, A. Pertsov, V. Slemzin, I. Tindo, M. Pakhomov, N. Salashchenko and O. Timofeev. "Instruments for imaging XUV-Spectroscopy of the Sun on board the CORONAS-1 satellite." In: Current Russian Research in Optics and Photonics, New Methods and Instruments for Space- and Earth-based Spectroscopy in XUV, UV, IR and Millimeter Waves, Igor Sobelman, Vladimir A. Slemzin, Editors, Proceedings of SPIE, V. 3406, pp. 1-19 (1998).

- 2) A. Ignatiev, N. Kolachevsky, V. Korneev, V. Krutov, S. Kuzin, A. Mitrofanov, A. Pertsov, E. Ragozin, V. Slemzin, I. Tindo, I. Zhitnik, N. Salashchenko and Roger J. Thomas. "Manufacture and testing of X-ray optical elements for the TEREK-C and RES-C instruments (the CORONAS-1 mission). In: Current Russian Research in Optics and Photonics: New Methods and Instruments for Space- and Earth-based Spectroscopy in XUV, IR and Millimeter Waves, Igor I. Sobelman, Vladimir A. Slemzin, Editors, Proceedings of SPIE, V. 3406, pp. 20-34 (1998).
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Report on the Project'98

# **Research and Development of Silicon Carbide and Related Materials**

APPENDIX A

Principal Investigator- Prof. V.E.Chelnokov  
Investigator-Doct.of Sc. A.A.Lebedev

Term of the Project: May 1998-April 1999

## Contents

Abstract of Project'98

Introduction

1. Conferences

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Department of Semiconductor Device Physics  
A.F. Ioffe Physico-Technical Institute  
Russian Academy of Sciences

## **Abstract of Project'98**

Subject of Research: Silicon Carbide and Related Materials,  
May 1998 - April 1999

Research has been conducted in three main directions:

1. Investigations in the field of single-crystal SiC, epitaxial methods for growth of SiC layers, fabrication of p-n structures on the basis of these layers. In addition, fundamental properties of SiC have been studied. 20 papers have been published in this field.
  2. Considerable attention has been given to research on carbon and diamond-like films, including nanocrystal diamond films, tetrahedral amorphous carbon films, amorphous silicon carbide, and various other amorphous materials. A number of techniques have been developed for growing films of this kind and heterostructures based on them have been fabricated. 15 papers published.
  3. Investigations of single-crystal GaN, epitaxial techniques for its growth, and fundamental properties of GaN. 7 papers published.
- In all, 42 papers have been published. Personnel of the Department participated in 7 international conferences and seminars.

## **Introduction**

In this report we give an account of the work done in the sixth stage (Project'98) of the Project "Research and Development of Silicon Carbide and Related Materials" (12 months from May 1998 to April 1999). Main research directions of Project'98 were summarized in Report-97. The research within the framework of Project'98 has been performed in the two following general directions: (1) investigation of single-crystal silicon carbide and III-V nitride and (2) investigation of related materials, including amorphous silicon carbide and diamond-like films. The total number of papers is 42.

## **1. Conferences**

**I. 1998 HIGH TEMPERATURE ELECTRONIC MATERIALS, DEVICES AND SENSORS CONFERENCE**

**FEBRUARY 22-27, 1998/BAHIA HOTEL, SAN DIEGO, CALIFORNIA USA**

**II. E-MRS CONFERENCE, SPRING MTG, JUNE 16-19, 1998 STRASBURG, FRANCE**

**III. ECSCRM'98**

**2<sup>ND</sup> EUROPEAN CONFERENCE ON SILICON CARBIDE AND RELATED MATERIALS**

**SEPTEMBER 2-4, 1998-MONTPELLIER, FRANCE**

**IV. DIAMOND 1998**

**9<sup>TH</sup> EUROPEAN CONFERENCE ON DIAMOND, DIAMOND-LIKE MATERIALS, NITRIDES AND SILICON CARBIDE**

**13-18 SEPTEMBER 1998, CRETA MARIS HOTEL, CRETE, GREECE**

**V. 1998 INTERNATIONAL SEMICONDUCTOR CONFERENCE**

**OCTOBER 6-10, 1998, SINAIA, ROMANIA**

**VI. FOURTH INTERNATIONAL HIGH TEMPERATURE ELECTRONICS CONFERENCE (HITEC)**

**ALBUQUERQUE MARRIOTT, ALBUQUERQUE, NEW MEXICO USA, JUNE 14-18, 1998**

**VII. SILICON CARBIDE MATERIALS AND DEVICE RESEARCH IN RUSSIA HOSTED BY THE GEORGIA TECH RESEARCH INSTITUTE AND THE DEPARTMENT OF THE NAVY**

**MARCH 5, 1998, ARLINGTON, VA**

## **2.Publications**

### **Papers and Abstracts**

#### **1. CAPACITANCE SPECTROSCOPY OF DEEP CENTRES IN SiC**

**A.A. Lebedev and N.A. Sobolev**

A.F. Ioffe Physico-Technical Institute 194021 St. Petersburg, RUSSIA

*Material Science Forum Vols.258-263 (1997) pp. 715-720 1997 Trans Tech Publications, Switzerland*

Keywords: Deep centres, DLTS, Silicon carbide, luminescence, structural defects.

Abstract. In the present work the parameters and distribution of deep centres in 6H and 4H SiC epitaxial layers and pn junctions grown by sublimation in an open system and by CVD were investigated. The deep level ionization energies and hole and electron capture cross-sections were determined by the DLTS and i-DLTS techniques. The possible structure of the observed centres and its influence on polytype transformation is discussed.

#### **2. HIGH POWER SiC- DEVICES. NEW RESULT AND PROSPECTS.**

**A.A. Lebedev and V.E.Chelnokov.**

A.F. Ioffe Physico-Technical Institute 194021 St. Petersburg, RUSSIA

(0-7803-4437-5/98/\$10.00 1998 IEEE)

Abstract. In the present work some theoretical analysis of SiC parameters important for electronic devices producing will be made. Also comparably description of the different technological methods using for sic growth will be done: for substrates growth - Lely method and modified Lely method; for n-type epilayers growth - sublimation epitaxy (SE), container free liquid phase epitaxy (CFLPE), chemical vapour deposition (CVD); for pn junction producing - SE, CFLPE, CVD, Al implantation (ID) and boron diffusion; for mesa structures formation -plasma-ion etching.

#### **3.CALCULATION OF THE SCHOTTKY BARRIER HEIGHT AT THE EARLY STAGE OF FORMATION OF THE (SILICON CARBIDE)-(METALLIC SUBMONOLAYER) CONTACT**

**S. Yu. Davydov, A. A. Lebedev, and S. K. Tikhonov**

A.F. Ioffe Physico-Technical Institute 194021 St. Petersburg, RUSSIA

(Submitted June 5, 1997; accepted for publication June 24, 1997)

Fiz. Tekh. Poluprovodn.32. 68-71 (January 1998)

Abstract. The position of the local and quasi-local states of metal atoms (alkali metals, group-III metals, the copper group) adsorbed on the surface of 6ff-SiC are calculated within the framework of the extended Anderson-Halstane model of the semiconductor

density of states. The results of these calculations are compared with the experimental data on Schottky barriers.

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#### 4. CURRENT-VOLTAGE CHARACTERISTICS OF GaN AND AlGaN *p-i-n* DIODES

**N.I. Kuznetsov**

A.F. Ioffe Physico-Technical Institute 194021 St. Petersburg, RUSSIA

K. G. Irvine

**Cree Research Inc. Durham, North Carolina 27713, USA**

(Submitted July 1, 1997; accepted for publication July 14, 1997)

Fiz. Tekh. Poluprovodn. 32, 369-372 (March 1998)

**Abstract.** The current-voltage characteristics of GaN and  $\text{Al}_{0.08}\text{Ga}_{0.92}\text{N}$  *p-i-n* diodes were investigated. The experimental *p-i-n* structures were grown by MOCVD on 6H-SiC with Si and Mg as dopants. The *i* region was formed by simultaneously doping with donor and acceptor impurities during growth. Analysis of the current-voltage characteristics showed that current flow in the *p-i-n* diodes is due to either drift of thermally excited holes or electron-hole recombination in the *I* region via impurity centers—just as predicted by the Ashley-Milnes theory. These impurity centers are attributed to Mg acceptor levels.

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#### ABSTRACTS OF CONFERENCE V

##### 5. HIGH TEMPERATURE TI-AL-BASED OHMIC CONTACTS TO P-6H-SiC

**A. N. Andreev, M. G. Rastegaeva, A. I. Babanin, M. A. Yagovkina, S. V. Rendakova,  
N. S. Savkina**

A.F. Ioffe Physico-Technical Institute 194021 St. Petersburg, RUSSIA

**Abstract.** Low-resistance ohmic contacts ( $10^{-4}$  Ohm  $\text{cm}^2$  at  $N_a - N_d = 10^{19} \text{ cm}^{-3}$ ) based on Ti-Al composition were prepared to p-6H-SiC grown by various techniques. These contacts demonstrate excellent reproductivity and the ones can operate at the environment temperatures up to 550°C and densities of direct current up to  $10^4 \text{ A/cm}^2$ . Structure and composition of contact coating were studied by AES and SIMS layer-by-layer technique and X-ray phase analysis. Advanced study of electrical characteristic of Ti-Al-based ohmic contacts was carried out.

##### 6. STRUCTURAL DEFECTS AND DEEP ACCEPTORS IN SILICON CARBIDE.

**Alexander. A. Lebedev**

A.F. Ioffe Physico-Technical Institute 194021 St. Petersburg, RUSSIA

**Abstract.** In the present work the parameters and distribution of deep centres in 6H and 4H SiC epitaxial layers and pn junctions grown by sublimation in an open system (SE) and by chemical vapor deposition (CVD) were investigated. The deep level ionization energies and hole and electron capture cross - sections were determined by deep level transient spectroscopy (DLTS). The possible structure of the observed centres and its influence on polytype transformation is discussed.

## **7. STEADY-STATE LIFETIME OF THE NONEQUILIBRIUM CARRIERS IN PROTON IRRADIATED 6H-SiC pn STRUCTURES**

**A.M.Strel'chuk<sup>a,b)</sup>, V.V.Kozlovski<sup>c)</sup>, N.S.Savkina<sup>b)</sup>, M.G.Rastegaeva<sup>b)</sup>,  
A.N.Andreev<sup>b)</sup>.**

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**Abstract.** The effect of proton irradiation on the value of the steady state lifetime of the nonequilibrium carriers in 6H-SiC epitaxial pn structures was investigated. The lifetime was determined as parameter of the Sah-Noyce-Shockley model, which was used for interpretation of the forward currents at current densities  $10^{-6} < J_M < 10^0$  A/cm. The irradiation dose  $3.6 \cdot 10^{14} \text{ cm}^{-2}$  decreased the lifetime of nonequilibrium carriers for deep-level recombination in the space charge region by up to 2 orders of magnitude. The irradiation dose of  $1.8 \cdot 10^{15} \text{ cm}^{-2}$ , or anneal in the range 300-800 K. did not change the lifetime.

## **8. STUDY OF NI-BASED OHMIC CONTACTS FABRICATED ON N-da-SiC POLAR FACES**

**M.G-Rasteegeva, A.N.Andreev, A.I.Babanin, E.N.Mokhov, V.I.Sankin**  
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**Abstract.** Electrical characteristics and composition of interface region were studied for Ni-based ohmic contacts prepared on the n-6H-SiC polar faces at the various annealing temperatures. Specific contact resistance as well as composition of interface region depends strongly on annealing temperature and surface orientation. The data are reported on dependence of specific contact resistance on uncompensated impurity concentration.

Fabricated ohmic contacts show stable electrical characteristics at the environment temperatures up to 550°C and current densities up to  $10^4$  A/cm<sup>2</sup>.

## 9. NEW RESULT IN 6H SiC "SITE-COMPETITION" EPITAXY

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**Abstract.** Silicon carbide (SiC) has received increasing attention as a wide bandgap semiconductor materials for high temperature electronics. For device application, it is important to control background doping in SiC epilayers. Under a fixed growth conditions, background doping strongly depends on the C/Si ratio of source gases. Furthermore, it is necessary to know the influence of the growth parameters. With an increase of C/Si ratio in gas phase there is a tendency to decrease concentration  $N_D-N_A$  up to  $10^{15}$ - $10^{13}$  cm<sup>-3</sup> [1]. The variation of concentration in n-type SiC layers is commonly done in the gas phase. The larger the carbon content the lesser amount of nitrogen into the composition of SiC and the lower is the concentration  $N_D-N_A$ . This replacement has been named "site competition"[2].

In this work we report on an investigation of 6H-SiC epitaxial layers grown by using atmospheric pressure CVD with a CH<sub>4</sub>-SiH<sub>4</sub>-H<sub>2</sub> gas system. The epilayers are characterized by volt-capacitance measurements and preliminary results of I-DLTS spectroscopy.

## CONFERENCE II

### 10. DOPING OF 6H-SiC pn STRUCTURES BY PROTON IRRADIATION

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V.V.Kozlovski.

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**Abstract.** The influence of proton irradiation on current-voltage characteristics,  $N_D-N_A$  value and parameters of deep centers in 6H-SiC ( $N_D-N_A = 4 \cdot 10^{16}$  cm<sup>-3</sup>) grown by sublimation epitaxy has been studied. Irradiation with 8 MeV protons was performed on a MGTs-20 cyclotron at doses in the range  $10^{14}$ - $10^{16}$  cm<sup>-2</sup>. First, irradiation with doses lower than  $3 \cdot 10^{14}$  leaves practically unchanged the resistance of pn structures. Second, with increasing irradiation dose, the resistance and degree of compensation in samples increases. DLTS studies have shown that irradiation causes, in addition to an increase in the concentrations of R-centre and Z1/Z2 centre previously observed in CVD-grown 6H SiC layers, formation of new centres in the lower half of the forbidden gap and also increases the concentration of D centre. Partial annealing of radiation and also partial restoration of electrical parameters on pn structures subjected to proton irradiation occurs after annealing at  $T \sim 500^\circ\text{C}$ . Finally, irradiation with doses  $> 5.4 \cdot 10^{15}$  cm<sup>-2</sup> results in the very high resistance of the forward biased pn structures and resistance remain high



even after heating to 500°C . These investigations show that radiation doping is promising way to obtain high resistivity silicon carbide layers. This work was partly supported by Arizona University and Schneider Group Research centre.

## 11. NEW RESULT IN OS SiC "SITE-COMPETITION" EPITAXY.

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**Abstract.** The investigation of 6H-SiC layers grown. by CVD in methane-silane-hydrogen system shows that with increase of C/Si .ratio in gas phase there is the inversion of type of conductivity. I-DLTS study of grown p-type conductivity layers shows that a main contribution to Na-Nd gives a deep acceptor centers, which are close to parameters of usual background centers in layers prepared by sublimation epitaxy method.

## 12. FUTURE TRENDS IN SiC-BASED MICROELECTRONIC DEVICES

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E. Garfunkel et al. (eds.), Fundamental Aspects of Ultrathin Dielectrics on Si-based Devices, 431-445.

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**Abstract.** Because of the large bandgap (3.0 eV for 6H SiC and 3.2 eV for 4H SiC) and radiation hardness, silicon carbide is prospective material for high temperature microelectronic devices. In SiC power devices unit switching power and speeds exceeding those in silicon by an order of magnitude or more are obtainable, with operating temperatures up to 1000 °C.

In the present work, a theoretical analysis of SiC parameters important for electronic devices producing will be made. Also a comparative description of different technological methods used for SiC growth will be done: for substrates growth Lely method and modified Lely method; for n-type epilayer growth sublimation epitaxy (SE), container free liquid phase epitaxy (CFLPE), and chemical vapor deposition (CVD); for p-n junction production - SE, CFLPE, CVD, Al implantation (ID) and boron diffusion; for mesa structures formation - plasma-ion etching.

Currently SiC diodes with breakdown voltage about 4.5 kV [1] have been realised. However, such SiC p-n junctions usually have very small working area  $< 10^{-3} \text{ cm}^2$ . Therefore to decrease defect density in SiC epilayers or to increase working area of the devices is one of the main problems in developing SiC power electronics.

Another problem is surface protection of manufactured devices. On the one hand, it is necessary to decrease surface breakdown, and on the other - to cover mesa-structure by a dielectric, with the critical electric field strength more than that for air. In this paper, we will try to generalize experience, which has been obtained in our laboratory during investigation of different SiC diodes, prepared by different technologies.

E. Garfunkel et al. (eds.), Fundamental Aspects of Ultrathin Dielectrics on Si-based Devices, 431-445.

### 13. OHMIC CONTACTS TO P-GaN

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**Abstract.** Electrical and structural characteristics of Pd contacts to p-GaN epitaxial layers were investigated. The contacts were deposited by thermal vacuum evaporation and treated at temperatures up to 600 °C. It was found that the Pd-p-GaN ohmic contacts (OCs) were formed at the samples temperature treatment of 600 °C. The dependence of the specific contact resistance on Na-Nd concentration was linear in the log scales in the range from  $3 \cdot 10^{17}$  to  $3 \cdot 10^{18} \text{ cm}^{-3}$ . On epitaxial layers with  $\text{Na-Nd} = 3 \cdot 10^{18} \text{ cm}^{-3}$  the contact resistance of  $2 \cdot 10^{-3} \text{ Ohm cm}^2$  has been measured. Pd-p-GaN OCs were stable at dc current densities up to  $14 \text{ kA/cm}^2$  and temperature 400 °C.

### 14. EXCITONIC ELECTROLUMINESCENCE OF 6H-SiC P-N STRUCTURES OBTAINED BY SUBLIMATION EPITAXY.

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(Submitted January 30, 1997; accepted for publication April 22, 1997)

Fiz. Teor. Poluprovodn. 31, 1347-1349 (November 1997)

**Abstract.** The electroluminescence spectra of 6H-SiC *p-n* structures obtained by sublimation epitaxy have been investigated. It has been found that the intensity of the excitonic band increases rapidly with increasing direct current density, and that this band dominates the emission spectrum of the diode at high direct-current densities and elevated temperatures. Investigation of the shift of the maximum of this band with increasing temperature shows that this band is most likely due to the recombination of a free exciton.  
© 1997 American Institute of Physics. [S1063-7826(97)01511-1]

### 15. GALLIUM NITRIDE MULTIOPERATE OPTOELECTRONIC DEVICES

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Abstract. Opportunity of creation of several optoelectronic devices such as optron. photoreceiver, switch device, IR-to-visible signal transformer and others based on i-n-GaN light emitting diodes (LED) is shown. Technology and properties of GaN LED in relation to the desired device properties are discussed. A discussion of issues relating to electrical and optical positive feed back between the device elements is also included.

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## **Conference VII**

### **16. SILICON CARBIDE MATERIALS AND DEVICE RESEARCH IN RUSSIA**

**V. Chelnokov, A. Lebedev, and V. Dmitriev**

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Abstract. Even though this report reviews studies in the field of SiC carried out all over Russia, emphasis is placed on investigations performed at the Ioffe Physico-Technical Institute, Russian Academy of Sciences, and in particular in our Laboratory. There are only two more groups in St. Petersburg which had also started investigations of SiC more than 40 years ago. These are the group of Professor Yu.M. Tairov (St. Petersburg University of Electrical Engineering), in the first place, and the group of Professor Yu.A. Vodakov (Ioffe Institute).

The research conducted in the group of Prof Yu.M. Tairov is mainly concerned with growth of SiC single crystals by modified sublimation method and the fundamental properties of such crystals. It should be noted that it was Professor Yu.M. Tairov who proposed more than 30 years ago the modified sublimation method, originally named Tairov's method.

Investigations of the group headed by Professor Yu.A. Vodakov are centered around developing a sublimation method for preparing epitaxial layers by sublimation and subsequent diffusion of various impurities to obtain p-n SiC structures. These structures have been used to fabricate LEDs with different wavelengths of emitted light. Among investigations of the fundamental properties of SiC, mention should be made of highly interesting results on polytypism in SiC.

There are also smaller groups with rather narrow scope of investigations of SiC. For example, interesting studies of ion implantation in SiC are being performed at the Kurchatov Institute of Atomic Energy. As far as we know, certain investigations have been and are being performed in Kiev (Ukraine) and Novosibirsk (Russia) and, possibly, at some other research centers of Russia and CIS countries.

However, at present we possess no information about these groups, since scientific communications are practically cut off and publications are scarce.

This work was supported in the part by Department Of Defence.

#### **Conference IV.**

ABSTRACT for 9th European Conference on Diamond, Diamond-Like Materials, Nitrides and Silicon Carbide, September 13-18, 1998, Crete, Greece.

#### **17. Pd OHMIC CONTACTS TO P-SIC 4H, 6H AND 15R POLYTYPES.**

**E-V. Kalinina\***, G.F. Kholujanov, N.S. Savkina, N.I. Kuznetsov, A.I. Babanin,  
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M.A. Yagovkina, and A.V. Shchukarev,  
Mekhanobr-Analyt. Institute, St. Petersburg, Russia.

#### **18. OPTICAL OBSERVATION OF BONDED AND QUASI-FREE HYDROGEN DIAMOND-LIKE CARBON.**

V.I. Ivanov-Omskii, M.P. Korobkov., B.R. Namozov, E.A. Smorgonskaya,  
S.G. Yastrebov  
A.F. Ioffe Physico-Technical Institute 194021 St. Petersburg, RUSSIA

#### **19. COPPER-ASSISTED DIAMOND NUCLEATION IN AMORPHOUS CARBON.**

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A.F. Ioffe Physico-Technical Institute 194021 St. Petersburg, RUSSIA

#### **Conference III**

#### **20 NEW RESULTS IN SUBLIMATION GROWTH OF THE SIC EPILAYERS**

N.S. Savkina, A.A. Lebedev, D.V. Davydov, A.M. Strelchuk, A.S. Tregubova,  
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#### **21. 6H-SIC SITE COMPETITION EPITAXY IN SILAN-METHAN-HYDROGEN GAS SYSTEM.**

V.V.Zelenin , A.A.Lebedev, M.G.Rastegaeva, D.V.Davidov, V.E.Chelnokov  
and M.L.Korogodskii.

A.F. Ioffe Physico-Technical Institute 194021 St. Petersburg, RUSSIA

## 22. INFLUENCE OF PROTON IRRADIATION ON RECOMBINATION CURRENT IN 6H-SiC pn STRUCTURES

A.M.Strechuk<sup>a,b)</sup>, V.V.Kozlovski<sup>c)</sup>, N.S.Savkina<sup>b)</sup>, M.G.Rastegaeva<sup>b)</sup>,  
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## 23. STUDIES OF THE EFFECT OF PROTON IRRADIATION OF 6H SIC PN JUNCTIONS

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Proceedings of the 7th International Conference on Silicon Carbide, III-Nitrides and  
Related Materials,  
Stockholm, Sweden, September 1997

## 24. CRYSTAL STRUCTURE AND OPTICAL PROPERTIES OF BULK GAN CRYSTALS GROWN FROM A MELT AT REDUCED PRESSURE

V. Sukhoveyev<sup>1,2</sup>, V. Ivantsov<sup>1,2</sup>, A. Zubrilov<sup>1,2</sup>, V. Nikolaev<sup>1</sup>, I. Nikitina<sup>1,2</sup>  
V. Bougrov<sup>1,2</sup>, D. Tsvetkov<sup>1,2</sup> and V. Dmitriev<sup>1,3,4</sup>

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<sup>4</sup>Material Science Research Center of Excellence, Howard University.  
2300 Sixth St., NW Washington DC, 20059, USA

Abstract. Crystal structure and optical properties of gallium nitride crystals grown from  
Ga-based melt at reduced pressure were investigated. The crystals were grown at the  
ambient gas pressure less than 2 atm at 1000°C. Lateral growth rate was about 1 mm/ht.

X-ray measurements showed that the crystals were 2H-GaN and had basal plane orientation. Photoluminescence and optical absorption measurements at various temperatures were performed. Optically pumped stimulated emission from bulk GaN crystals at room temperature was observed.

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## **25. HIGH QUALITY 6H- AND 4H-SIC PN STRUCTURES WITH STABLE ELECTRIC BREAKDOWN GROWN BY LIQUID PHASE EPITAXY**

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**Abstract.** Material issues currently limiting performance of silicon carbide devices include high defect density in epitaxial structures, insufficient doping level in contact layers, and unstable electric breakdown. In this paper, we review recent results on SiC liquid phase epitaxy (LPE) addressing these problems.

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## **26. DISSOLUTION AND GROWTH OF SILICON CARBIDE CRYSTALS IN MELT-SOLUTIONS**

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**Abstract.** Dissolution and growth of bulk SiC crystals in molten transition metals and their silicon-rich alloys were studied. Bulk plate-like SiC crystals up to 7 mm in lateral size and 0.2 mm in thickness have been successfully grown without seed from the selected molten alloy with a lateral growth rate of the order of 1mm/hr. Normal growth rate was significantly less than lateral one. Optical and electron microscopical observations revealed that the crystals are free of micropipes.

X-ray diffraction showed an alteration of polytypes from 6H for crystals spontaneously grown on the melt surface to 6H+15R. or 21R. for crystals grown in the melt volume, and 3C or 3C+6H for crystals formed on the bottom of the graphite crucible. Auger electron

spectroscopy and secondary ion mass spectrometry did not detect any traces of melt components in the crystals (within the accuracy of 0.01 at.‰). Photoluminescence of the grown crystals was measured.

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## **27. AEROSOLS**

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### **27.1 IR-ACTIVE MODES OF FULLERENE GROWN ON SILVER**

**V.I.Ivanov-Omskii, E.K.Kuznetsova, S.G.Yastrebov and G.A.Dyuzhev**  
A.F. Ioffe Physico-Technical Institute 194021 St. Petersburg, RUSSIA

### **27.2 DIAMOND NANOCLOUDS NUCLEATION IN AMORPHOUS CARBON MEDIA**

**V.I.Ivanov-Omskii, S.G.Yastrebov**  
A.F. Ioffe Physico-Technical Institute 194021 St. Petersburg, RUSSIA

## **28. BICRYSTALLOGRAPHY OF TILE EPITAXIAL SYSTEMS III-V NITRIDES ON SAPPHIRE: THEORY AND EXPERIMENT**

**A. N. Efimov, A. A. Lebedev and A. M. Tsaregorotsev**  
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*(Received 27 March 1997; accepted 3 September 1997)*

**Abstract.** Orientation relationships (OR) for gallium, aluminium or indium nitrides on sapphire substrates have been systematically studied, both theoretically and experimentally, as a function of substrate orientation. Using an approach developed in this paper, all variety of published ORs have been classified into a few types. It is shown that the dependence of ORs on both sapphire cut orientation and the layer compound cannot be recognized within the framework of the commonly accepted coincidence site lattices concept. Nevertheless, the dependence of ORs on the layer compound for (0001) and (1120) sapphire substrate has been successfully described, without using any fitting parameters, by symmetry analysis as proposed previously by the authors and computer Monte Carlo simulation of the initial stage of epitaxy. The experimental part of this paper consists of obtaining the nitride layers on sapphire substrate with various orientations and examining the ORs, taking into account the direction of the polar sixfold axis, by both conventional X-ray and Kossel line techniques. As a result, for the first time the ORs on all close-packed cuts of sapphire (with polarity) have been determined and a new type of OR for aluminium nitride has been found. All experimental data agree with theoretical considerations.

J. Appl. Cryst. (1998). 31, 000-000

## 29. BONDED AND NON-BONDED HYDROGEN IN DIAMOND-LIKE CARBON

V.I. Ivanov-Omskii, M.P. Korobkov, B.R. Namozov, E.A. Smorgonskaya,  
S.G. Yastrebov

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**Abstract.** The techniques of infrared absorption and vacuum ultraviolet Raman spectroscopy are applied to study the states of hydrogen in diamond-like a-C:H. It is shown that, along with the state covalently bonded with carbon, there exists a quasi-free state of stretched H<sub>2</sub> molecules adsorbed by graphite-like structural fragments. A model of thermally-induced reversible transfer of hydrogen between the bonded and non-bonded states is suggested to describe the experimental results of thermal treatment. The techniques of infrared absorption and vacuum ultraviolet Raman spectroscopy are applied to study the states of hydrogen in diamond-like a-C:H. It is shown that, along with the state covalently bonded with carbon, there exists a quasi-free state of stretched H<sub>2</sub> molecules adsorbed by graphite-like structural fragments. A model of thermally-induced reversible transfer of hydrogen between the bonded and non-bonded states is suggested to describe the experimental results of thermal treatment. © 1998 Elsevier Science B.V. All rights reserved.

Journal of Non-Crystalline Solids 227-230 (1998) 627-631

## 30. CARBON CLUSTERS IN AMORPHOUS HYDROGENATED CARBON

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**Abstract.** Direct observation is reported of diamond cluster nucleation resulting from the thermalisation of accelerated carbon atoms (ions) during growth of hydrogenated amorphous carbon films by ion sputtering of graphite. Transmission electron microscopy (TEM) (100 keV), selected area electron diffraction (SAED) and optical absorption (1 to 6 eV) were used to study the prepared hydrogenated amorphous carbon films. The size-distribution function of diamond crystals derived from TEM images is considered in terms of the fluctuation theory. To investigate the graphite-like constituent of a-dH films, the optical data are analyzed under the assumption that the fundamental absorption edge is due to nanometer building blocks constructed of a set of sp<sup>2</sup>-bonded carbon atoms assembled together as six-membered units embedded in an amorphous matrix. A mechanism of diamond crystal nucleation is considered.

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Journal of Non-Crystalline Solids 227-230 (1998) 622-626

## 31. DIAMOND AND DIAMOND-LIKE FILM APPLICATIONS. PROCEEDINGS THIRD INTERNATIONAL SIMPOSIUM ON DIAMOND FILMS

ST.PETERSBURG, RUSSIA, JUNE 16-19, 1996



TECHNOMIC PUBLISHING CO, INC., LANCASTER-BASEL.

### 31.1 PRODUCTION AND PROPERTIES OF LOW-TEMPERATURE TETRAHEDRAL CARBON FILMS

**O.I Konkov, E.I. Terukov and I N. Trapeznikova**

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Abstract. Films of high-tetrahedral amorphous carbon have been produced by glow discharge decomposition of methane in Ar/H<sub>2</sub> at temperatures of about 300 K. Structural, optical and electron transport properties of asdeposited films have been investigated. Optically transparent amorphous carbon films with refractive indices, 1.5-2.2 and density 2.75-2.8 g/cm<sup>3</sup> were obtained. The concentration of (optically) active hydrogen atoms was about 21-28 at.%. The sp<sup>2</sup>/sp<sup>3</sup> ratio ranged between 0.8-0.85. The optical bandgap was 3-3.6 eV, electron mobility <2 cm<sup>2</sup>/V s, and the resistivity 0.01-10<sup>16</sup> Ohm cm at 300 K. Photoluminescence spectra had a wide intensity band with a maximum at 487-493 nm and a half-width of 0.6-0.8 eV.

### 31.2 NUCLEATION AND GROWTH OF ANISOTROPIC CLUSTERS

**V.I. Siklitsky**

A.F. Ioffe Physico-Technical Institute 194021 St. Petersburg, RUSSIA

Abstract. The influence of the probability that spherical particles are inter-connected during the nucleation and growth of fractal cluster is considered in analyzing cluster formation in Diamond Like Carbon (DLC).

### 31.3. KEATING-HARRISON MODEL FOR WIDE BAND GAP SEMICONDUCTOR ELASTIC PROPERTIES DESCRIPTION

**S. Yu. Davydov and S. K. Tikhonov**

A.F. Ioffe Physico-Technical Institute 194021 St. Petersburg, RUSSIA

Abstract. On the basis of the direct comparison of deformation schemes in Keating and Samson models the expressions relating force constants to crystal covalence, covalent energy and nearest neighbor distance have been obtained. Also the numerical calculations of elastic coefficients for the cubic and hexagonal modifications of diamond, silicon carbide and nitrides of B, Al, and Ga have been performed.

### 31.4 ON THE CALCULATION OF DIELECTRIC AND OPTICAL PROPERTIES OF WIDE BAND GAP SEMICONDUCTORS

**S. Yu. Davydov and S. K. Tikhonov**

A.F. Ioffe Physico-Technical Institute 194021 St. Petersburg, RUSSIA

Abstract. The bond orbital model has been used to calculate linear and quadric dielectric susceptibility, electro-optical coefficient and static permittivity for diamond and cubic modification of SiC, BN, AlN, and GaN.

### **31.5 AMORPHOUS CARBON: FREE AND BOUND HYDROGEN**

**I.N.Kapitonov, O.I Konkov, I N. Trapeznikova and E.I. Terukov**  
A.F. Ioffe Physico-Technical Institute 194021 St. Petersburg, RUSSIA

Abstract. We have studied the effusion of hydrogen and other gases from amorphous carbon films while annealing in the temperature range 100-1000 °C. The amount of hydrogen in the material (28 at. % has been determined by the direct method. The amount of free hydrogen in the investigated material was determined as about 4 at. %. The temperature dependence of free hydrogen effusion had a maximum at 400°C. A wide band of hydrogen effusion was observed at temperatures greater than 400°C. The amount of hydrogen evolved after weak C-H bond breaking was of the order 10 at. % The hydrogen amount evolved after strong C-H bond breaking was 12 at.% in these a-C:H films.

### **31.6 EPITAXIAL AND HETEROEPITAXIAL GROWTH OF SILICON CARBIDE ON SiC AND ALN/AL<sub>2</sub>O<sub>3</sub> SUBSTRATES**

**A. N. Kuznetsov, A. A. Lebedev, M. G. Rastegaeva,  
E. V. Bogdanova and E.I. Terukov**  
A.F. Ioffe Physico-Technical Institute 194021 St. Petersburg, RUSSIA

Abstract. SiC films have been grown on SiC and AlN/Al<sub>2</sub>O<sub>3</sub> substrates by reactive magnetron sputtering of a silicon carbide target in an Ar/CH<sub>4</sub>/SiH<sub>4</sub> mixed plasma. Crystal perfection was investigated as a function of substrate temperature in a temperature range 700-1400 K. The SiC epaayers surface were made into Schottky diodes by deposition of molybdenums films. Current-voltage and capacitance-voltage measurements showed an n-type conductivity with uncompensated donor concentration of  $5 \cdot 10^{18} \text{ cm}^{-3}$ . The barrier high was 1.0 eV.

### **31.7 ROOM-TEMPERATURE PHOTOLUMINESCENCE OF AMORPHOUS HYDROGENATED SILICON CARBIDE DOPED WITH ERBIUM**

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